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Review

A preliminary study of acid volcanic rocks for stonemeal application

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ABSTRACT

This study is related to the chemical and mineralogical characterizations and to the availability of mineral nutrients in four fresh samples of fine material generated in crushing mills of volcanic rocks of the Serra Geral Formation in northeastern state of Rio Grande do Sul (RS), Brazil. The objective of the study is to estimate the agronomic potential of rocks, after pulverization, to its application as a fertilizer for the soil by means of the incorporation of rock, using a technique known as stonemeal. The analytical techniques were used: X-ray diffraction (XRD), X-ray fluorescence (XRF), mass spectrometry with inductively coupled plasma (ICP-MS), and leaching tests by extraction with citric acid solution diluted to 2%. The results indicated the potential of providing nutrients to the soil, corroborating the results of other studies based on the technique stonemeal. The following characteristics were identified in the samples: presence of calcium and magnesium as carbonates; high alkalinity; good availability of phosphorus; potassium average availability; and of the presence of micronutrients such as zinc, boron, copper, iron, and manganese.

Contents

1. Introduction

Stonemeal, soil remineralization, and petrofertilization are terms used to describe the technique of soil fertilization with the application of rock reject (Machado et al., 2009). The application of rock reject in agriculture is very old. Leonardos (1976a) reported centuries-old cases of the application of rock fragments in soil. In Brazil, many studies have been developed in recent years with the application of rock reject in soils, with positive results for productivity (da Silva et al., 2011; Lourenço, 2011; Prates, 1998; Plewka et al., 2009). In 2009, the First Brazilian Congress of Stonemeal was held, with the aim, among others, to present the advancements

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of research about stonemeal in Brazil (EBPA, 2009). In 2013, the II Brazilian Congress of Stonemeal emphasized the stimulation of technological research and the development of the potential of the use of rocks dusts as mechanisms of remineralization and rejuvenation of degraded soils, a practice that has become reality in many sustainable farming systems in various regions of the country.

de Souto et al. (2011), after Petersen and Almeida (2008), discussed the environmental feasibility of using rock reject in agroecological practices associated with other forms of management. Stonemeal also promotes positive effects on soil quality, assisting in the recovery and conservation of soils. In addition to minimizing the use of soluble fertilizers, stonemeal offers economic advantages in its application. In this context, da Silva et al. (2008), in experiments with Cerrado soil in a greenhouse, concluded that stonemeal showed positive effects on the chemical quality of the subsoil. Fyfe et al. (1983) showed that the application of approximately 1 t/ha/year rock powder with potassium (K^{+}), calcium (Ca^{2+}), and phosphorus (P^{5+}) can restore fertility in exhausted soils, such as some areas of the Cerrado. In a way, application of stonemeal is a type of sustainable fertilization that enables, over time, the rejuvenation of the agricultural soil with part recovery of their constituents without affecting its natural chemical balance. The use of rock reject provides benefits to the soil by promoting the increase of the cation exchange capacity (CEC) due to the formation of clay neominerals during the weathering process (Melamed et al., 2009). In spite of these advantages, Van Straaten (2006) warned that the technique may be disadvantageous if the added materials have low nutrient concentrations and low solubility, characteristics which can reduce the agronomic efficiency, especially in temperate regions.

The high degree of comminution of rock dusts generated in crushing of rock facilitates the exposure of minerals when the rocky matrix is microcrystalline, such as volcanic rocks, the focus in this study. Reducing the particle size increases the exposure of mineral phases and, consequently, the action of weathering which causes the mineral alteration, including clay minerals generation, and elements release in the dissolved phase, and increase in cation exchange.

Basalts are among the most studied rocks because they have the highest possibility of supplying nutrients to the soil, especially phosphorus, calcium, magnesium, and micronutrients, and they have a low silica content (Theodoro, 2011). The pulverized basalts used in soil remineralization can provide the macronutrients Ca^{2+} and Mg^{2+} and micronutrients or trace elements such as Cu^{2+}, Zn^{2+}, Mn^{2+}, Fe^{3+}, Co^{2+}, Ni^{2+}, and V^{4+} (Lapido and Ribeiro, 2009).

The rocks with horizontal fracture, sought for extraction of basalt slabs, are acidic and consist of fine- to medium-grained dacites and rhyodacites, with colors ranging from dark to light gray, and may have shades of green to brown. The basic and intermediate rocks consist of basalts and andesites with colors ranging from gray to dark gray, with predominantly solid structure and irregular and conchoidal fracturing (Prates, 1998).

Considering the various studies and reports on the positive effects of the use of rock reject in agriculture and environmental practices aimed at sustainability, as well as the potential of basaltic volcanic rocks as a source of raw material, we developed a research project to assess the characteristics of tailings from mining basalts of the Serra Geral Formation, produced in Local Productive Arrangement (APL) Basaltos Nova Prata, in northeastern RS, and the possibility of its use in stonemeal. The small local miners exploit the quarries for the sole purpose of producing aggregates for the construction industry.

The study region is known for the intensive production of cereal crops, vineyards, fruits and vegetables, and silviculture activity, all requiring periodic application of inputs for fertilization and soils correction, which originating from the weathering of basaltic matrix and conditioned by petrographic and topographic changes. According to Streck et al. (2002), in mountain areas, escarpment, and on top of hills in northeastern RS, there are three classes of soils: Oxisol in areas of hills on the edge of the plateau; ferric Chernosol Argilúvico in the valleys and profiles of strand of moderate steepness; and dystrophic Neossolo Lytic.

In this study, are presented the first results of chemical and mineralogical characterization of four samples of powdered basalt.

2. The state of the art

The stonemeal technique is defined as an agricultural practice of incorporating rocks and/or minerals to the soil, and the liming and natural phosphate particular cases of this practice (Leonardos, 1976b). In addition, as a kind of remineralization (Leonardos et al., 2000), in which the rock powder is used to rejuvenate poor soil or leachate, the pursuit of balance of fertility, conservation of natural resources, and naturally sustainable productivity.

In the 1960s, the Green Revolution, was deployed in Latin America, defending the idea of mass food production through manipulation of soil with intensive use of industrial inputs, irrigation, and farm mechanization. This movement was fueled by the risk of a global crisis in food production, the increase in world population, and a pessimistic outlook about the availability of food in the world (Almeida and Navarro, 1998). Since then, the consumption of agricultural inputs such as chemical fertilizers, has achieved prices that undermine the balance of the sector in Brazil, an agricultural and country dependent on imported raw material for fertilizers and low fertility soils, which are in mostly acididic and have widespread nutrient deficiencies, especially phosphorus and potassium (Sanches and Salinas, 1981).

In the 19801s, the German chemist Justus von Liebig, discovered NPK (nitrogen (N), phosphorus (P), and potassium (K)) and initiated the era of chemical fertilizers (Pinheiro, 2003; Maar, 2006). At the same time, Hensel (2014) proposed that powdered rocks have the same effect without unbalancing the environment, and at low cost. Only a few farmers recognized practice, and his book *Bread from Stone*, was censored (Pinheiro, 2003). In his book was reissued in 1997, taking advantage of concerns about the nutritional quality and food safety.

The use of rocks as recuperation sources of plant nutrients, soil reclaimer and refreshing, you can configure an alternative technology to assist in reducing the use of chemicals, especially embedded in highly soluble forms, such as the fertilizer formulations NPK (Pinheiro and Barreto, 1996; Theodoro, 2000).

Experiments and research on the use of powdered rocks were performed worldwide in the eighteenth century by James Hutton (Bakey, 1967). Considered the founder of the geological sciences, Hutton used loam and similar rocks on his farm in Scotland to increase soil fertility. Lacroix (1922) drew attention to the potential for nutrients contained in most rocks. In North America, Graham (1941) suggested the use of plagioclase as calcium source based on experimental data and Keller (1950) pointed out the potential of dozens of types of rocks as a source of potassium, calcium and trace elements, and have since been advocate the practice of stonemeal.

In Congo, the D' hotman de Vulliers (1947) recommended the use of powdered basalt rock for rejuvenation of depleted soils of humid regions. The study was based on a long series of field experiments, which showed a considerable increase in the production of cane sugar, and the cost of applying 71 t/recovered with increasing crop production after 4 ha. Evans (1947) showed an increase of 33.7% and 56.7% in dry matter production with the cultivation of oats in pots, applying powder basaltic rock in the proportions of 247 and 497 t/ha, respectively.

In Brazil, Il'chenko and Guimarães (1953) highlighted the potential of Cedro do Abaeté, Serra da Mata da Corda, and Poços de Caldas rocks in Minas Gerais. Fraya (1952) conducted surveys to assess phonolites, altered with high contents of K_2O, for use as rock powder to be applied to the soil as fertilizer. Kavaleridze (1978) reported that the predominant basaltic rocks in southern Brazil are rich in silica, calcium, magnesium, and potassium, recommending turning them into powder for use in stonemeal. Motta and Feiden (1992) found that the application of 40 t/ha of basalt powder was enough to raise the level of available phosphorus, behaving as a corrective fertilization of the soil. Kiehl (2002) noted that the use of basalt rock dust for soil amendment brought positive results, making it a favorable alternative to farmers, recommending use of 50–100 t of basalt powder per hectare in poor soils to make them fertile.

The rock powder, is a low cost material with decentralized production, is recommended for the remineralization of soils (Gillman, 1980). This technique is considered an alternative to reduce the use of industrial fertilizers in the soil. The stonemeal constitutes a source of nutrients for the plants grown for long periods and promotes increased cation exchange capacity of the soil due to the formation of new clay minerals during the process of mineral alteration (Melamed et al., 2005). The release of nutrients from the crystal lattice of the rocks occurs through the action of organic acids produced by plants and microorganisms in the soil (Moraes, 2014). Gilman (Melamed et al., 2005) and Gillman et al. (2001) illustrated the positive effects of the application of high concentrations of basaltic rock in soil with low fertility. In the case of Gillman (1980), after twelve months of incubation a significant increase in pH and cation exchange capacity was observed, with the effect more pronounced with decreasing particle size and increasing the contact time between the material and soil. Gillman et al. (2001) examined the behavior of soils from seven Queensland, sites incubated with different concentrations of particles of basalt (0.1, 5.25 and 50 t/ha), and observed significant increases in pH, exchange capacity cations, and the levels of alkali cations.

Escosteguy and Klant (1998) concluded that, due to the low release of nutrients from ground rocks, such materials cannot be used as the main source of nutrients for plants. They found also that, in general, small doses tested yielded increases in the potassium, calcium, magnesium, and pH. It is worth noting, however, that the use of rock dust is a practice of fertilization of soils whose results are obtained in the medium and long term, and its effects are more lasting than chemical fertilization, which should be applied necessarily in all crops. The rates of nutrient release happen very slowly (Blum, 1989) the effectiveness of the rock particles as a source of nutrients for the soil is questioned due to the low solubility and the need to apply large quantities of the same medium to give positive responses (Bolland and Baker, 2000) is due to the dissolution of the rock particles to be a very slow and complex process, depending on factors such as the chemical mineralogical composition, particle size, pH and biological activity of the soil, and the weather exposure (Osterroht, 2003).

However, alternative methods for soil fertilization, such as stonemeal and bio-organic fertilizers are essential. In addition, with that, improving the physico-chemical characteristics of the soil, the negative environmental impacts generated by the high-cost synthetic fertilizers that can cripple agriculture on small farms.

3. Materials and methods

3.1. Study area and sampling procedures

In the study area, located in Nova Prata Mining District, there are basic, intermediate, and acidic volcanic rocks. At the top and middle part of the plateau, acidic rocks are interspersed with basic and intermediate rocks. At the base of the plateau, there are only basic and intermediate rocks (Prates, 1998). Samples were obtained from Basel Industry and Trade Minerals (sample B1), Concresul Crushing (sample C1), Union of the Industry of Extraction Quarries of Nova Prata (sample NP2), and Zilli and Basalt Crushing (sample Z1). These results allow a preliminary assessment of the potential of the material sampled in stonemeal technique.

The samples belong to the Facies Caxias, and correspond to the acid volcanic effusions which are in the upper position of the volcanic effusion, in general. This lithology is described by Nardy et al. (2008) as belonging to Group Palmas of the Serra Geral Formation.

The preparation of the samples C1, Z1, and NP2 consisted of the following procedures: drying, quartering to obtain aliquots, pulverization, and screening of the aliquots. The drying followed the procedures of the standard (ABNT, 1986), the quartering was done in a Jones-type splitter; aliquots were pulverized in a Pulverisette 5 planetary ball mill, and, the sieving was done at 0.6 mm, with the passing material intended for analysis. The sample B1 just was subjected to sieving, because the sample was already in a powder state with a particle size less than 0.6 mm. The chosen particle size was based on preliminary tests of the extraction of nutrients with citric acid solution diluted to 2%, where the granulometry of 0.6 mm showed better results in the liberation of nutrients.

3.2. Mineralogical and chemical analysis procedures

Granulometric, mineralogical, and chemical analyses were performed. Particle size analyzes were performed at the Soil Laboratory of Center for Environmental Studies, University Center La Salle (UNILASALLE), and at the Laboratory of Mineral Processing (LAPROM), Federal University of Rio Grande do Sul (UFRGS). For the coarse fraction, a set of sieves with different openings was used in a vibrating machine. For the fine material (particle size less than 500 μm), a Silas laser diffraction granulometer was used. The semi-quantitative mineralogical analysis was performed at the Laboratory of X-ray Diffraction of UFRGS, with a Siemens diffractometer, Model Bruker – AXS D5000. The data were processed using DiffracPlus® Siemens – Bruker – AXS, version 11 software.

The mass spectrometry with inductively coupled plasma (ICP-MS) chemical analyses were performed at Acme Analytical Laboratories Ltd. (Acmelabs), Vancouver, Canada, and SGS Geosol Laboratories Ltda., Belo Horizonte.

The analysis of nutrients in aqueous media was performed at the Laboratory of Soil Science, Faculty of Agronomy of UFRGS. For this analysis, only the less than 0.15-mm NP2 sample was used.

Aliquots of similar samples to those sent to the Soil Laboratory of the Faculty of Agronomy were tested for the extraction on the Chemistry Laboratory UNILASALLE, using the methodology described by Theodoro et al. (2010), which uses citric acid solution diluted to 2% for the extraction. The use of citric acid in the nutrient-release study aimed to simulate the environment soil/root and promoting conditions similar to the natural environment. Organic acids, especially citric acid, are commonly exuded by the roots of the plants and remain in high concentration in the rhizosphere (Song and Huang, 1988). These acids have a high ability to interact with metals to form metal–organic complexes in the soil solution, inducing the release of nutrients by soil minerals (Martin and Sparks, 1983; Melo et al., 1995). In the extraction by citric acid method, 1.0 g of sample was transferred to a 250-mL Erlenmeyer flask and 100 mL of citric acid solution diluted to 2% was added. The solution was stirred for 30 min, about 30–40 oscillations per minute. After this time, samples were vacuum filtered with 0.45-mm membrane filter and preserved with HNO_3 and then sent to the Analytical Center of Embrapa Temperate Climate for analysis of calcium, copper, iron, magnesium, manganese, potassium, and zinc.

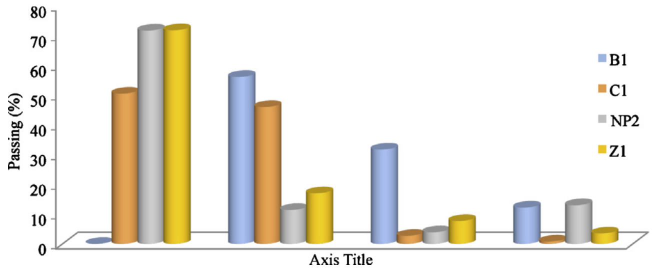

Fig. 1. Histograms of particle size distribution of the *in situ* samples in four granulometry ranges.

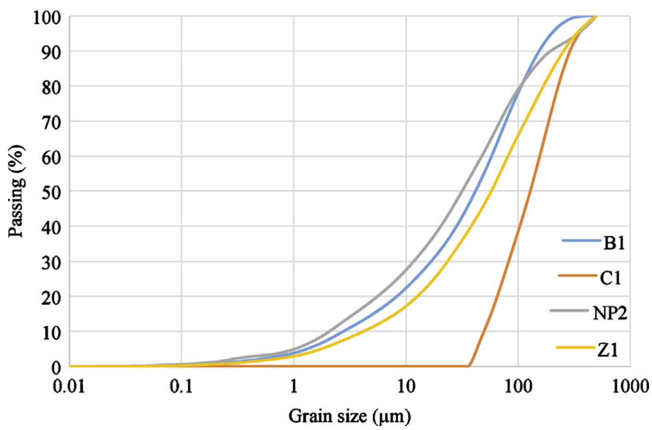

Fig. 2. Granulometric distribution curves of the <500-μm fraction obtained by laser diffraction in the four samples.

4. Results and discussion

4.1. Granulometric distribution

Fig. 1 shows the histograms of particle size distribution of the *in situ* samples in four granulometry ranges. These data show that residues from the extraction of basalt in the study region have different particle sizes, but all have a predominant particle size in the range of fine sand and silt; this influences the surface area, exposure of minerals, and, therefore, the release of nutrients.

Fig. 2 shows granulometric distribution curves of the <500-μm fraction obtained by laser diffraction in the four samples. Only the sample Z1, with no clay fraction, has a different behavior; the other samples show similar behavior.

4.2. Mineralogical analysis

Table 1 shows the mineralogy of the crystalline phase of the sample, as identified by X-ray diffraction (XRD), and their frequencies in the four samples. The technique used does not detect amorphous content present in the rock. According to Nardy et al. (2008), the amorphous content present in the Palmas type rock is around 63%.

Table 1
Predominant minerals of crystalline phase in the B1, C1, NP2, and Z1 acid volcanic rock powder samples.

Mineral	B1	C1	NP2	Z1
Labradorite	73.5	51.0	52.0	61.0
Quartz	9.0	19.0	15.0	13.0
Augite	6.0	–	12.0	13.0
Alkali feldspar	5.0	15.0	–	–
Smectite	3.0	7.0	10.0	–
Barite	2.0	3.0	4.0	4.0
Hematite	1.0	1.0	2.0	2.0
Heulandite	0.5	–	–	–
Gypsum	–	4.0	–	–
Kaolinite	–	–	5.0	7.0

Table 2
Potential (%) reactivity (climatization) minerals of the crystalline phase in the B1, C1, NP2, and Z1 acid volcanic rock powder samples.

Mineral class	B1 (%)	C1 (%)	NP2 (%)	Z1 (%)
Non-reactive	9.0	19.0	15.0	13.0
Slightly reactive	3.0	4.0	11.0	13.0
Reactive	84.5	66.0	64.0	74.0
Very reactive	3.5	11.0	10.0	0.0

Table 3
Nutrient concentrations in the B1, C1, NP2, and Z1 acid volcanic rock powder samples.

Element	B1 (%)	C1 (%)	NP2 (%)	Z1 (%)
Al	1.25	0.83	1.93	0.52
Ca	1.1	0.7	0.6	0.8
Fe	2.9	3.05	3.67	2.62
P	0.1	0.1	0.1	0.1
Mg	0.3	0.3	0.3	0.3
Na	0.4	0.22	0.14	0.12
K	0.3	0.2	0.2	0.1
	B1 (mg/dm^3)	C1 (mg/dm^3)	NP2 (mg/dm^3)	Z1 (mg/dm^3)
B	4	3	3	2
Co	46	7	27	7
Cu	51	49	70	50
S	<200	<200	<200	<200
Mn	388	351	1076	381
Mo	0.4	1	0.7	1
Ni	3	4	6	3
Zn	48	49	57	44

The identification of the labradorite as dominant plagioclase indicates the calcic chemical affinity of the studied rocks. In addition, certain dosage of potassium in the sample C1, due to the relatively high concentration of alkali feldspar. The low amount of quartz in sample B1 in relation to other samples is probably related to the segregation of the coarser mineral grains during the beneficiation process, limiting its occurrence in <0.6-mm waste. Gypsum detected in the sample C1 may be contamination from the concrete plant near the collection point. Kaolinite and smectite are common clay mineral derivatives of the alteration of plagioclases.

Table 2 shows potential reactivity of crystallines minerals in the samples. In this study was considered the following categories: non-reactive minerals (quartz and hematite); slightly reactive (barite and kaolinite); reactive (labradorite, augite, and alkali feldspar); and very reactive (smectite and heulandite).

4.3. Chemical analysis

The chemical results obtained in the present study show that the samples consist mainly of iron and aluminum. These results are consistent with the data obtained from mineralogy, indicating the presence of amorphous oxides, labradorite, augite, feldspar, and smectite in greater amounts in the samples. In addition, the samples have some macro and micronutrients known to be relevant to the development and maintenance of plants (Malavolta, 2006). These results only indicate the presence of nutrients; the liberation and reactivity are independent variables. Table 3 shows the concentrations of macro and micronutrients contained in the samples obtained by ICP-MS analysis.

Table 4 shows the results for the oxides. The compositional contents are similar for the four samples, considering that it is samples processed and subjected to segregation of minerals and, based on their location and classification of Nardy et al. (2008),

Table 4
Major oxides (%) in the B1, C1, NP2, and Z1 acid volcanic rock powder samples.

Oxide	B1	C1	NP2	Z1
Al_2O_3	13.2	13.1	13.7	13.0
BaO	0.1	0.1	0.1	0.1
CaO	3.9	3.5	2.7	3.9
Fe_2O_3	7.0	6.9	7.5	7.0
K_2O	3.6	3.8	3.3	3.5
MgO	1.4	1.4	1.2	1.5
MnO	0.1	0.1	0.2	0.1
Na_2O	3.3	3.2	2.7	3.3
P_2O_5	0.3	0.3	0.2	0.3
SiO_2	65.1	66.0	63.8	65.1

Table 5
Available nutrients in aqueous medium in samples of B1, C1, NP2, and Z1 acid volcanic rock powder samples.

Nutrient	B1	C1	NP2	Z1
Al ($_{cmolc}/dm^3$)	Nd	Nd	Nd	Nd
Ca ($_{cmolc}/dm^3$)	6.7	7.0	8.1	7.8
Mg ($_{cmolc}/dm^3$)	1	1	2.2	0.9
Mn (mg/dm^3)	3	2	6	2
P (mg/dm^3)	>100	>100	>100	>100
K (mg/dm^3)	152	59	104	76
B (mg/dm^3)	0.2	0.2	0.2	0.2
Cu (mg/dm^3)	8	4	2	5
Zn (mg/dm^3)	2	2	2	2

Nd: not detected.

Table 6
Agronomic characteristics identified in B1, C1, Z1, and NP2 acid volcanic rock powder samples.

Agronomic characteristic	B1	C1	NP2	Z1
pH	7.7	8.6	6.7	8.6
CEC (c_{molc}/dm^3)	8.8	8.9	11.6	9.5
(%) clay	12.0	6.0	51.0	6.0

these samples can be classified as acidic volcanic rocks. The oxides are dominated by SiO_2, due to the predominance of silicates and aluminosilicates.

4.4. Analysis of availability of nutrients

Table 5 shows available nutrients in the volcanic rock powder samples and Table 6 shows some physical and chemical parameters of agronomic quality. Even if the samples have high levels of aluminum oxide, as detected in the XRF study, Table 5 shows that this element is not released into the aqueous medium. Aluminum is toxic to plants and can cause the reduction of their growth (Malavolta, 2006). Phosphorus, calcium, and zinc showed available values concentrations considered "high" for soils, according to Malavolta's (Malavolta, 2006) classification. Based on Malavolta (2006), potassium is considered "high" just for sample B1, while magnesium and boron showed average values.

Continuous tests and long times may better assess the ability of these materials to maintain the levels of availability nutrients observed. According to Theodoro et al. (2010), rocks may take many years to release their nutrients into the medium.

Other promising results were the high pH of the samples (between 6.7 and 8.6), which does not exacerbate the normal condition of soil acidity of the study area, and the absence of aluminum (Al^{3+}) because this element is toxic and inhibitory of cation exchange capacity of clay minerals.

Although the nutrient values are significant in the samples, the CEC values are low, including the sample NP2, with high clay content (51%) and 11.6 $cmol_c/dm^3$ of CEC. Unlike clayey soils, which have surface negative charges, the samples possess primary

Table 7
Nutrient content in B1, C1, NP2, and Z1 acid volcanic rock powder samples.

Element	B1 (mg/dm^3)	C1 (mg/dm^3)	NP2 (mg/dm^3)	Z1 (mg/dm^3)
Al	0.43	0.41	0.40	0.39
Ca	2.37	1.19	2.82	0.11
Mg	0.07	0.03	0.13	0.10
Cu	0.06	0.14	0.11	0.14
Fe	3.88	4.18	6.01	5.76
Mn	0.87	0.46	2.00	1.81
K	5.40	3.60	1.90	1.10
Zn	0.03	0.10	0.10	0.07

comminuted minerals with low negative charge in the surface. The CEC values found in the samples of rock powder are due to the presence of kaolinite and smectite (Table 1).

The availability of some macronutrients and micronutrients were evaluated in conditions close to natural through nutrient extraction with citric acid solution diluted to 2% (Theodoro et al., 2010). Table 7 presents the results of this extraction for some elements.

The extraction with dilute citric acid 2%, showed an available potassium content lower than those extracted in the soil procedure analysis, the latter using strong acids such as HCl and H_2SO_4. The best results in the phosphorus extraction of samples were NP2 and B1.

The tests with dilute citric acid 2%, as expected, did not coincide with the results of the classic agronomic analysis. The extractions Ca^{2+}, Mg^{2+}, and K^+ solution with 2% citric acid showed a reduction by 10–1000 times with respect to extraction by agronomic analyses, while for Cu and Zn, the results were similar in both methods.

5. Conclusions

In general, the advantages of using waste rocks consist in correcting the pH in nutrient supply and its long residual effect. On the other hand, a disadvantage is the slow weathering and, therefore, the slow release of nutrients to the plants.

Considering that the rocks that have been tested as alternatives to replacing nutrients in cultivated soils are complex composition of different particle size and use different effects depending on the characteristics of the environment where they will be applied (soil, plants, and organisms), there are several challenges to be overcome by research.

The studies of nutrient availability to the aqueous medium indicated that all samples released macronutrients and micronutrients important for plant growth. Other relevant data obtained in this study was the unavailability of aluminum to the aqueous medium, which is a great advantage, because it is a toxic element for plant development.

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